

TECHNOLOGY UTILIZATION

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METALLURGICAL PROCESSING

A COMPILATION



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

## Foreword

The National Aeronautics and Space Administration and the Atomic Energy Commission have established a Technology Utilization Program for the rapid dissemination of information on technological developments which have potential utility outside the aerospace and nuclear communities. By encouraging multiple application of the results of their research and development, NASA and AEC earn for the public an increased return on the public investment in aerospace and nuclear R&D programs.

The items in this compilation, all relating to metallurgical processing, are presented in two sections. The first section includes processes which are general in scope and applicable to a variety of metals or alloys. The second describes the processes that concern specific metals and their alloys. This latter section is divided according to metals in alphabetical order.

Additional technical information on individual devices and techniques can be requested by circling the appropriate number on the Reader Service Card enclosed in this compilation.

Unless otherwise stated, NASA and AEC contemplate no patent action on the technology described.

We appreciate comment by readers and welcome hearing about the relevance and utility of the information in this compilation.

Jeffery T. Hamilton, *Director*  
*Technology Utilization Office*  
*National Aeronautics and Space Administration*

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# Section 1. General

## MOLDING PROCEDURE FOR CASTING A VARIETY OF ALLOYS

In a recent procedure for preparation of molds, the sand and the sodium silicate binder are blended in a suitable mixer until all silicate lumps have disappeared (5 to 10 minutes). The ester is added and blended into the mixture (about 1 minute). The composition is placed in the molding flask containing the pattern. The composition starts to set within 30 minutes and is hard within 2 to 3 hours. At the end of this period, the molds are heated at 120°C (250°F) for 2 hours to eliminate any moisture, and the pattern is removed. The cope and drag are closed prior to pouring metal.

Castings which have been made using this molding procedure are in the 0.5 to 50 kg (1-100 pound) range

with section thickness of 0.3 to 6.6 cm (1/8 to 3 in.). The castings are machined to achieve proper finish and dimensions.

Castings of radiographic quality have been produced from the following alloys: 309 stainless steel, 17-4PH stainless steel, Waspaloy PWA-652, Hastelloy GMR-235D, Inconel 625, Aluminum 195, 356, KO-1, Berylco 33-20C, and Navy Bronze. Many other alloys could be used with this process.

Source: D. Kourtides, M. J. Fontes, and

E. R. Leibfritz

Ames Research Center

(ARC-10358)

*No further documentation is available.*

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## LEVITATION-MELTING TECHNIQUE FOR METALS AND ALLOYS

Levitation melting is a recent technique for melting metallic materials by suspension of the dense charge in space with an electromagnetic field. The main advantage of this method for preparing alloys is that a completely homogeneous melt can be produced quickly.

Two new coil configurations were developed that permit a wide variety of metals to be levitated successfully in the molten state. Although only limited quantities of material may be processed with the present coils, the quantities are adequate for research purposes.

A helium-gas-quenching method was developed for this technique to minimize contamination and segregation. The helium gas is precooled with liquid nitrogen to provide a faster quenching rate. Aluminum and pure metals with higher melting points, such as zirconium, titanium, and vanadium, have been quenched to solid state by this technique.

It was found that the levitation-melting technique is ideal for the preparation of alloys for Mössbauer studies, which require the addition of a small quantity of a specific isotope. For example, small quantities of Fe<sup>57</sup> were successfully added to vanadium and palladium to produce a homogeneous distribution of the isotope.

The following documentation may be obtained from:

National Technical Information Service

Springfield, Virginia 22151

Single document price \$6.00

(or microfiche \$0.95)

Reference:

ANL-7398 (N68-27205), Levitation Melting of Metals and Alloys.

Source: J. W. Downey

Metallurgy Division

Argonne National Laboratory

(ARG-10240)

## IMPROVED RETORT FOR CLEANING METAL POWDERS WITH HYDROGEN

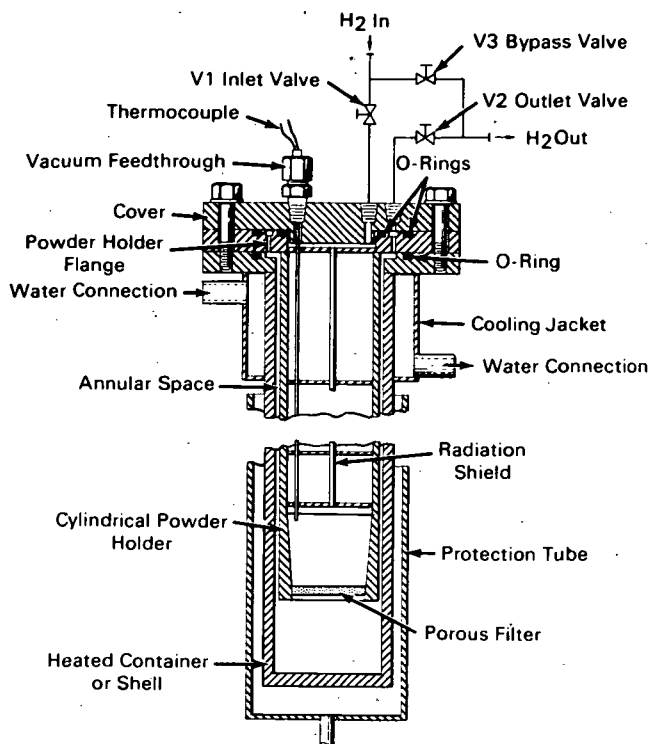


Fig. 1. Retort Assembly

Figure 1 shows an improved retort for cleaning metal powders with hydrogen. The retort consists of a heated container or shell housing a cylindrical powder holder, a radiation shield, and an optional porous filter. Other parts of the apparatus include a thermocouple, a vacuum feedthrough threaded into the cover and through holes in the radiation shield, three valves, three "O" rings, and a cooling jacket with water connections.

During operation, hydrogen enters the retort through the inlet valve, flows down, around, and through openings in the radiation shields, through the powder bed on top of the porous filter, through the filter, up the annular space between the shell and powder holder, through holes in the powder holder flange, and exits through the outlet valve. The hot gas exhausting through the annular space preheats the incoming gas flowing down the powder holder through the radiation shield and gives a long hot zone with very uniform temperature.

Figure 2 depicts an alternate method of cleaning

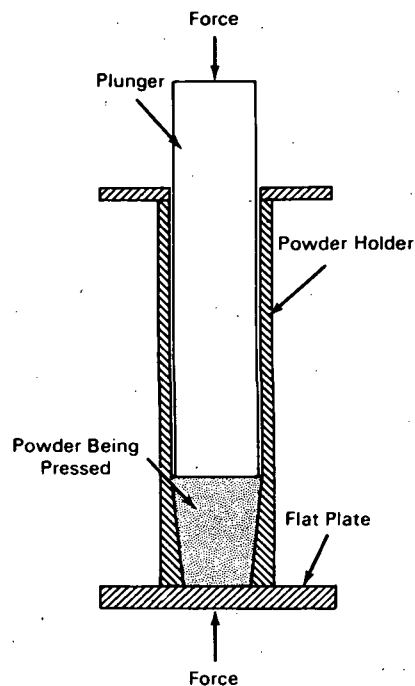


Fig. 2. Pressing Powders in Powder Holder

powders. After removal of the metal filter shown in Figure 1, cold pressing of the powder occurs in the tapered section of the powder holder. A plunger applies pressure through the top of the powder holder. The tapered design of the holder and the pressure differential across the bed from top to bottom prevent channeling between the powder holder and powder bed.

The following documentation may be obtained from:

National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$6.00  
(or microfiche \$0.95)

### Reference:

NASA TN-D-4912 (N-69-10827), Feasibility of Producing Dispersion Strengthened Chromium by Ball-Milling in Hydrogen Halides.

Source: A. Arias  
Lewis Research Center  
(LEW-10718)

## ULTRASONIC METAL ETCHING FOR METALLOGRAPHIC ANALYSIS



Experiments have indicated that selective metal etching for metallographic analysis may be achieved by an ultrasonic etching method. The ultrasonic etch delineates microstructural features which may not be discernible in specimens prepared for metallographic analysis by standard chemical etching procedures.

A magnetostrictive transducer was used to generate ultrasonic vibrations which were transmitted to a layer of distilled water separating the transducer head from the metal specimen. Cavitation bubbles in the ultrasonically excited water produced preferential damage (etching) of the metallurgical phases or grain boundaries, depending on the hardness and toughness of the phase or structure of the metal specimens. The materials studied were the unalloyed metals, zinc, nickel 270, iron, and tantalum, and the nickel-base alloy Udimet 700.

An electron microscope replica of the surface of the latter, which was subjected to cavitation in water at room temperature for two hours, is shown in the illustration.

The following documentation may be obtained from:

National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$3.00  
(or microfiche \$0.95)

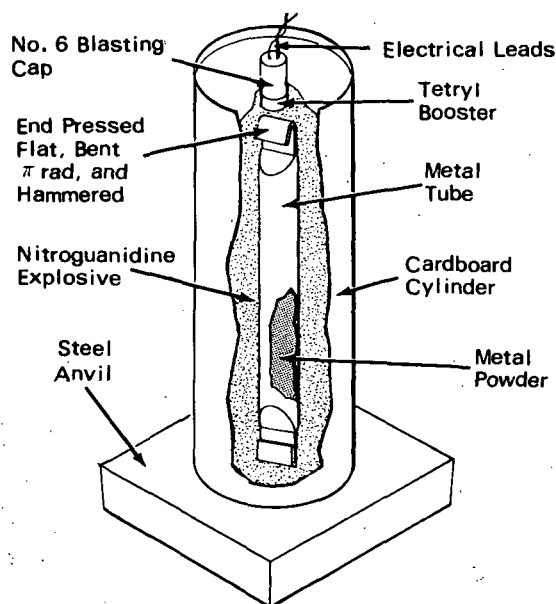
Reference:

NASA TN-D-6014 (N70-38568), Study of Cavitation Damage to High-Purity Metals and a Nickel-Base Superalloy in Water.

Source: S. G. Young  
Lewis Research Center  
(LEW-11230)



## UNIQUE INTERMETALLIC COMPOUNDS PREPARED BY SHOCK WAVE SYNTHESIS



A new materials fabrication technique uses explosion-generated shock waves to compress a mixture of fine ground metallic powder beyond the crystal fusion point.

This method requires mixing high purity metal powder particles in the atomic proportions to produce the desired intermetallic compound. The mixed powders are then introduced into a metal tube which is closed, but not sealed, at each end so that gases generated during explosive densification and/or com-

paction can be expelled. This metal tube is then inserted into a cardboard cylinder and surrounded with an explosive, as shown in the figure. The cylinder can be made of any material strong enough to contain the explosive powder while retaining its cylindrical shape up to the time of detonation.

After the cylinder and tube are loaded and assembled, an electric detonator (centrally positioned in one end of the cylinder) is activated. Upon reaching the inner metal tube containing the metal powders, the explosive front continues as an annular pressure wave which develops into a high-velocity conical shock-wave having pressures in excess of  $10^{10}$  N/m<sup>2</sup>. This overcomes the bonds between the powder particles and results in high densification, grain strength, and deformation of the particles. The resultant powder compact produced is, in effect, pressure quenched as a function of the high rate of pressure decay, i.e., the superficial particle melting is quenched and re-solidified in microseconds.

Source: G. Otto and O.Y. Reece  
Marshall Space Flight Center  
and U. Roy of  
University of Alabama  
under contract to  
Marshall Space Flight Center  
(MFS-20861)

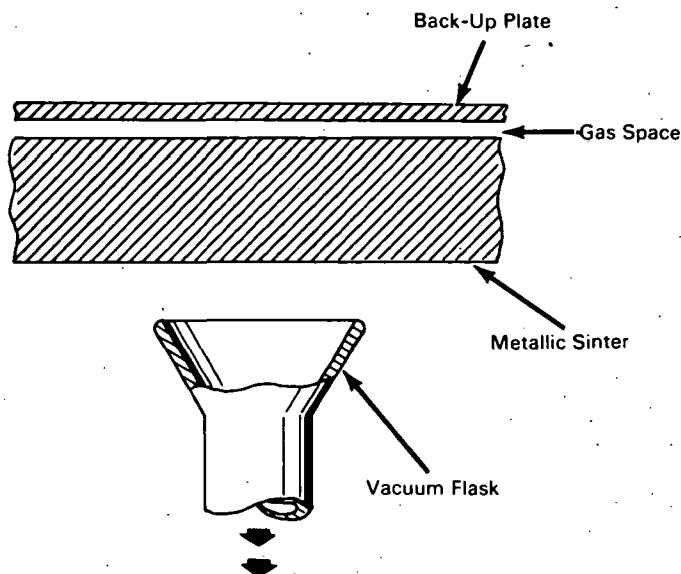
*No further documentation is available.*

## FUEL CELL LIFE IMPROVED BY METALLIC SINTER ACTIVATION

To improve the service life of fuel cell electrodes, metallic sinter is welded before its activation to the metal back-up plate of the electrode, as shown in the figure. One side of the sinter is attached to a sheet metal back-up plate by means of, but not limited to, electron beam welds. The activation procedure consists of cleaning the sinter, passing an activator solution through the sinter, and recleaning the sinter. The initial electrode cleaning is done by soaking for 5

minutes in deionized water, and then removing the entrapped water by applying a vacuum (from the open end of a vacuum filter flask) to the exposed side of the sinter. The activator solution (including the corrosion inhibitor), is allowed to flow into an opening between the sinter and back-up plate surfaces (e.g., the gas passage area in fuel cell assemblies). The solution is forced through the sinter either by gravity or by a

## METALLURGICAL PROCESSING



vacuum applied to the exposed face of the sinter, as was done in the initial cleaning operation.

The relative amount of sinter activation is noted by the change in sinter color. If the color is near black, a longer action time is used before applying the vacuum. The electrode is then cleaned by forcing deionized water to flow through the sinter into a flask. The residue collected in the vacuum operation is weighed, and this weight is subtracted from the

initial weight of activator particles to give the weight of the activator entrapped by the metallic sinter. The electrode is then dried at  $115 \pm 20^\circ\text{C}$  ( $235 \pm 35^\circ\text{F}$ ) for two hours.

Source: W. A. Taylor of  
Pratt and Whitney Corp.  
under contract to  
Manned Spacecraft Center  
(MSC-10965)

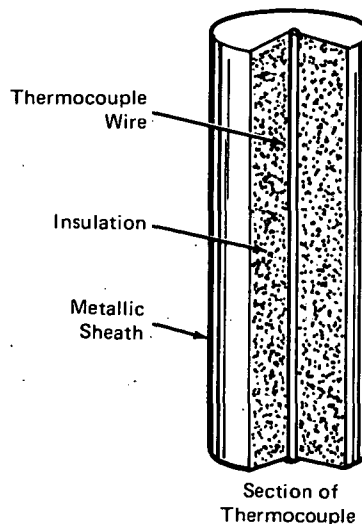
*Circle 1 on Reader Service Card.*

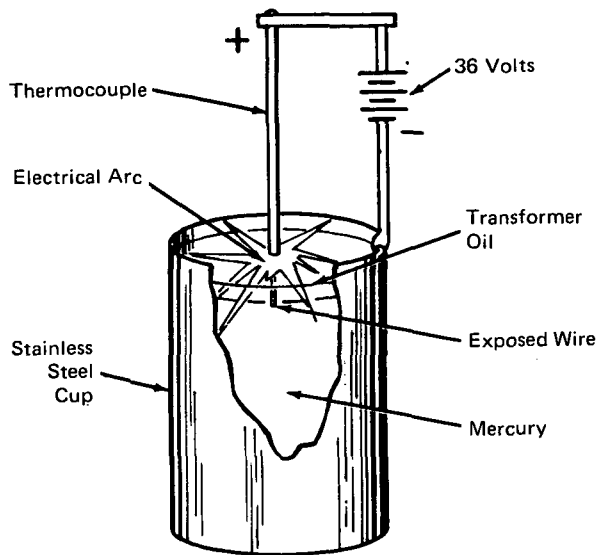
## ARC RAPIDLY REMOVES METALLIC SHEATH FROM THERMOCOUPLES

To determine whether there are any deteriorations in the thermocouple structure, it is necessary to remove the protective metal sheaths enclosing the thermocouples. This process is efficiently accomplished with an arc.

The equipment for sheath removal includes a stainless steel beaker containing mercury with a thin layer of transformer oil on top as shown in the figure. The beaker is connected to the negative terminal of a 36 V dc supply; the positive lead is connected to the thermocouple sheath by a spring-loaded clip.

As the thermocouple is gradually lowered into the mercury, an arc is initiated at the first point of con-





tact. The sheath is sputtered off at this interface leaving the electrically neutral thermocouple leads intact. This process continues until the entire thermocouple is immersed.

Because the oil layer retains fumes, the described process may be operated remotely or by hand. However, this work should be conducted in a hood having a forced air ventilation.

Source: R. Park of  
Astronuclear Laboratory  
Westinghouse Corp.  
under contract to  
Space Nuclear Systems Office  
(NUC-10021)

*No further documentation is available.*

## Section 2. Specific Metals

### PROCESS FOR PREPARING DISPERSIONS OF ALKALI METALS

For a convenient production of highly pure, finely divided alkali metal particles (e.g., sodium and potassium), the alkali metal is dissolved directly from the solid at a relatively low temperature in a solution of the complexing agent and the preferred solvent. For example, N-ethyl carbazole when dissolved in tetrahydrofuran, dissolves sodium in essentially equimolar quantity at approximately  $-70^{\circ}\text{C}$ , and the pure metal is quantitatively precipitated as the solution approaches room temperature.

The size and uniformity of the particles of the precipitated sodium are controllable over an appreciable range by variation of the rate of temperature change and the amount of agitation during precipitation. The precipitate can be removed by filtration,

and the filtrate can be reused to dissolve more metal in a continuous process. The finely divided metal may be redispersed in a conventional manner in a suitable nonreactive liquid (e.g., petroleum ether).

During solution of the alkali metal, commonly occurring solid impurities are readily removed by filtration in a preliminary step before precipitation of the alkali metal. If the original metal should contain an impurity that is soluble at the low temperature of the alkali metal reaction, such impurity is retained in solution during precipitation of alkali metal.

Source: A. Rembaum and R. F. Landel  
Jet Propulsion Laboratory  
(JPL-734)

*Circle 2 on Reader Service Card.*

## MIXED ETHER BATH FOR ELECTRODEPOSITION OF ALUMINUM

When anisole is added to a Brenner aluminum plating bath, the electrodeposits have greater physical strength and the bath has a lower vapor pressure. This improved mixed ether bath consists of anisole to ethyl ether (volume ratio 1:2); aluminum chloride (molar concentration 3.4M); and lithium aluminum hydride (molar concentration 0.3-0.4M). The ether content makes the bath very hazardous; in addition, the bath reacts with oxygen, carbon dioxide, and moisture; therefore, the plating is prepared in a dry nitrogen atmosphere within a glove box.

The mixed ether is cooled during the addition of anhydrous aluminum chloride ( $\text{AlCl}_3$ ) by an immersed cooling coil through which a coolant is circulated.

The  $\text{AlCl}_3$  is slowly dissolved into the cold, mixed ether so that the bath temperature does not exceed 303K. After the solution is prepared, it is undisturbed for 16 to 24 hours to permit settling of

the minute grey suspended particles. A medium-porosity, fritted-glass Buchner filter is then used to produce the desired crystal-clear, light amber solution.

The complete plating solution is accomplished by slowly mixing the  $\text{AlCl}_3$ -mixed ether solution with the lithium aluminum hydride  $\text{LiAlH}_4$ -mixed ether solution. This addition of  $\text{LiAlH}_4$  must be carefully performed to prevent excessive foaming, excessive temperature (keep below 303K), and excessive localized precipitation. The bath is complete when the molar concentration of  $\text{LiAlH}_4$  is between 0.3 and 0.4M in the resultant solution and the molar concentration of  $\text{AlCl}_3$  is 3.4M.

Source: K. Lui of  
Electro-Optical Systems, Inc.  
under contract to  
Langley Research Center  
(LAR-10200)

*Circle 3 on Reader Service Card.*

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## A METHOD FOR OBTAINING HIGH DUCTILITY IN CRITICAL AREAS OF ALUMINUM CASTINGS

During proof-pressure testing of aluminum castings, cracks have appeared in areas where the wall thickness did not meet minimum requirements. It is thought that the variations occur during closing of the mold. A method has been developed for salvaging the material and reducing costs.

Wrought aluminum alloys are more ductile than cast aluminum alloys and provide a desirable, high-strength substitute in the damaged area. The brittle area is removed by machining and replaced by an identical section machined from wrought aluminum.

The wrought segment can be either welded to the cast alloy in the conventional manner or built in with a heat-treatable welding rod. After this operation, the entire casting is heat treated again to the required condition.

Source: R. A. Zuech and M. L. Strangeland of  
North American Rockwell Corp.  
under contract to  
Marshall Space Flight Center  
(MFS-18705)

*Circle 4 on Reader Service Card.*

### TREATMENT INCREASES STRESS-CORROSION RESISTANCE OF ALUMINUM ALLOYS

Near optimum values of the precipitation-hardened aluminum alloys (2000, 6000, and 7000 series), which provide both yield strength and resistance to stress corrosion, are achieved by heat treatment. The alloys are first heat treated at the recommended temperatures in accordance with the manufacturer's specifications. For example, aluminum alloy 7075 is solution annealed at 465 to 475°C (870-890°F) for 10 minutes to 1 hour, rapidly cooled to a temperature between 115 and 125°C (240-260°F), and aged from 23 to 28 hours at this temperature. This treatment converts the alloys to the T6 condition, which provides maximum yield strength but very poor stress-corrosion resistance. Although overaging of the T6 alloy to the T73 condition (by a proprietary process)

improves its stress-corrosion resistance, the gain is counteracted by a 14% decrease in yield strength. By explosively shock loading the alloy immediately after its conversion to the T73 condition, 80% of the yield strength loss is recovered without degrading the resistance of the alloy to stress corrosion. The shock loading level must be sufficient to produce moderate plastic deformation without significantly changing the dimensions of the alloy.

Source: A. J. Jacobs of  
North American Aviation, Inc.  
under contract to  
Marshall Space Flight Center  
(MFS-1840)

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### THERMAL STRESS-RELIEF TREATMENTS FOR 2219 ALUMINUM ALLOY

Residual stresses incurred in roll-forming of 13-by-69 cm-by-9 m diameter (5-by-27 in.-by-33 ft.) 2219 aluminum alloy Y-ring segments result in distortion of the Y-ring during subsequent machining. As a result, three theoretically promising thermal stress relief treatments were evaluated.

The treatments evaluated are post aging (175°C, 350°F, 9 hours) of full-scale and sub-scale parts formed in the aged T81 condition, stress relieving (225°C, 435°F, 3 hours) the full-scale and sub-scale parts formed in the aged T81 condition, and aging (175°C, 18 hours) of sub-scale parts formed in the unaged T31 condition.

Resistance to stress-corrosion cracking of a full-scale Y-ring segment formed in the aged T81 condition is improved by post aging at 175°C for 9 hours, but a decrease in short transverse ductility results.

After stress-relieving at 225°C for 3 hours, no stress-corrosion cracking is observed on specimens from a full scale Y-ring segment formed in the aged T81 condition. This treatment, however, results in a

decrease in average short transverse yield strength of approximately  $48.3 \times 10^6 \text{ N/m}^2$  (7000 psi).

Residual stress of sub-scale Y-ring segments formed in the aged T81 condition is reduced by post aging at 175°C for 9 hours or by stress-relieving at 225°C for 3 hours. A slight advantage is indicated for the stress-relief treatment. No appreciable degradation of long transverse mechanical properties is observed for either treatment. Restraining of parts during thermal treatments does not significantly affect either residual stress or long transverse mechanical properties.

The lowest residual stress obtained for sub-scale Y-ring segments forms in the unaged T31 condition and ages after forming. In this case, the long transverse mechanical properties are excellent. Residual surface stresses after aging are slightly higher for parts restrained during aging.

Source: Boeing Corp.  
under contract to  
Marshall Space Flight Center  
(MFS-1213)

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## METALLURGICAL PROCESSING

## IMPROVED THERMAL TREATMENT OF ALUMINUM ALLOY 7075

A newly developed tempering treatment considerably increases the corrosion resistance of the 7075-T6 aluminum alloy and concomitantly preserves its yield strength. The new treatment consists of the following steps:

- (1) The 7075-T6 alloy is heated for 1 hour at 493°C (900°F) and immediately quenched in oil at 115°C (240°F).
- (2) The alloy is then aged at 120°C (250°F) for 24 hours and then air-cooled to room temperature.
- (3) Finally, the alloy is overaged at 163°C (325°F) for a period of up to 18 hours.

The results of tests on samples of the alloy subjected to the above treatments show that when the overaging period is 12 hours (at 163°C), the alloy exhibits a yield strength of  $53 \times 10^7 \text{ N/m}^2$  (73,000 psi) and a time to failure of more than 1000 minutes in a controlled corrosive environment.

Source: F. H. Cocks of  
Tyco Labs, Inc.  
under contract to  
Marshall Space Flight Center  
(MFS-20083)

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## CRUCIBLE CAST FROM BERYLLIUM OXIDE AND REFRACTORY CEMENT

A method was developed for large crucibles which will not deteriorate in the presence of fused salt-molten metal mixtures. The initial mixture comprising 85 to 93% by dry weight of BeO and 7 to 15% hydraulic refractory cement is cast in the desired vessel shape and fired at 600°C. The refractory cement, which contains 3 or 4 of the components of alumina, magnesia, baria, strontia, or calcia, forms eutectic bonds with the BeO when fired at this temperature. These cement components are inert in the fused salt-molten metal environment. The cast and fired crucible has a 10 to 20% porosity, which is desirable for thermal shock resistance.

After the initial firing of the vessel, a coating of refractory oxides is applied to the vessel inner surfaces and fired to form eutectic phases with the BeO. Two coating compositions found effective with BeO and resistant to attack by the fused salt and molten metal contain the following compounds, in percentage by weight:

	Coating #1	Coating #2
Al <sub>2</sub> O <sub>3</sub>	28%	55%
BeO	27.5%	
MgO	44.5%	
CaO		35%
SrO		10%

The following documentation may be obtained from:  
U.S. Patent Office  
Single document price \$0.50

## Reference:

U.S. Patent Number 3,150,281.

Source: Z. D. Jastrzebski  
Chemical Engineering Division  
Argonne National Laboratory  
(ARG-22)

### POROUS MANDRELS PROVIDE UNIFORM DEFORMATION IN HYDROSTATIC POWDER METALLURGY

The gas pressure bonding process was investigated as a potential means for hot isostatic pressing of relatively large, complex beryllium machining blanks from beryllium powder. Because existing methods of pressing were not efficient, various types of copper powders were pressureless sintered as a means of producing porous mandrels.

On empirical grounds, hydrogen-sintering parameters of 2 hours at 870°C (1600°F) were chosen as the most promising for the desired density. In actual practice, pressureless sintering for 3 hours at 925°C

(1700°F) was found to give densities of 57 to 60 percent of the theoretical ones. The porous copper mandrels were placed in the forming jig and the beryllium powder arranged over and around them. Hot, isostatic pressing formed the blank, and the porous mandrels were removed by leaching.

Source: H. D. Hanes and P. J. Gripshover of  
Battelle Memorial Institute  
under contract to  
Marshall Space Flight Center  
(MFS-1972)

*Circle 8 on Reader Service Card.*

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### ELECTROCHEMICAL SINTERING PROCESS FOR PRODUCING ELECTRODES FROM CADMIUM FELT AND A NICKEL OR SILVER GRID

Commercially fabricated cadmium felt electrodes are now available for testing and evaluation. Cadmium felt is made by drawing off the solvent from a slurry of fine cadmium fibers by means of suction filtration. The layer of intertwined fibers on the fiber bed is then pressed to the desired thickness. Thermal sintering of the felt is impossible because heating at temperatures below the low melting point of cadmium does not result in permeation at contact points.

Two pieces of cadmium felt are sandwiched around a nickel screen or silver expanded metal grid, held together by mold compression, and electrochemically sintered by being put through several charge and discharge cycles at low current density (0.5-1.0 mA/cm<sup>2</sup> approximately). The pieces of felt grow together through the grid. A network of conductive current

paths forms gradually beginning at the grid and working out into the felt to more and more of active cadmium material. This causes the ampere-hour capacity of the electrode to increase. A strong finished product results.

The use of cadmium felt is a novel method of affixing active cadmium to electrode substrates. This felt serves a double function both as the active material and as the substrate. Advantages of the electrode production process are ease of quality control and speed.

Source: T. J. Hennigan, N. Potter, W. Webster,  
E. Colston, and J. T. Stemmle  
Goddard Space Flight Center  
(GSC-10764)

*No further documentation is available.*

## METALLURGICAL PROCESSING

**HIGH TEMPERATURE ALLOY**

A cobalt-based alloy (S-1) was made which is capable of being formed into various mill products (such as tubing and sheet); is capable of being welded; has good high temperature strength; and is not subject to embrittlement produced by high temperature aging.

By replacing part or all of the tungsten with molybdenum and by limiting the tungsten and carbon contents to less than 7 and 0.07 percents, respectively, the characteristics of this alloy (S-1) closely match the characteristics of prior art high-strength cobalt-based alloys. The alloy is notable, also, for the absence of any tendency to embrittle on heat aging above 540°C (1000°F).

Forming of a 12.7-mm (0.5-in) thick S-1 alloy plate into a cylinder was performed at room temperature. Welding and machining operations were per-

formed with an ease equal to that normally encountered with standard alloys.

The superior ductility retention of this alloy (S-1) became more clearly evident after aging for 1000 hours at 870°C (1600°F) when it was found that elongation had still remained at about 30 percent. By comparison, prior art high-temperature, cobalt-based alloys have an elongation of about 12 to 19 percent after aging for 100 hours and 6 to 11 percent after aging for 1000 hours.

Source: J. E. Semmel, Jr. and R. G. Frank  
of General Electric Corp.  
under contract to  
Lewis Research Center  
(LEW-10377)

*Circle 9 on Reader Service Card.*

**PROCESS YIELDS Co-Fe ALLOYS WITH SUPERIOR HIGH-TEMPERATURE MAGNETIC PROPERTIES**

The cobalt-iron alloys having superior high-temperature magnetic properties are prepared from ultrapure cobalt and iron. High quality cobalt and iron as the starting materials are refined by the electron beam, float zone method in a vacuum of  $10^{-6}$  Torr. After three zone passes, the impurities are reduced to less than 90 ppm. The binary alloys are then prepared by arc melting the predetermined proportions of the refined metals in purified argon. The total of all impurities in each batch of alloy obtained from the melts was less than 250 ppm.

Specimens of 5 alloys, which contained 6.4, 7.0, 8.0, 9.3, and 10.0 percent of iron, respectively, were prepared for tests. These specimens consisted of 0.3-mm thick strips which were cold rolled from 12-mm thick rods. During the cold rolling, only the 9.3- and 10.0-percent-iron alloys required intermediate annealing. All specimens were given a final stress relief at 1020°C, a temperature just below the Curie point.

The cobalt-iron alloys containing from 7.0-to-9.3-

percent iron prepared by this method have the highest Curie point of all known magnetically soft materials. Their high permeability, low hysteresis loss, good saturation induction, and square loop characteristics make them suitable for power transformers, rotating machinery, and square-loop devices in the temperature range from 600 to 1000°C.

The following documentation may be obtained from:

National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$6.00  
(or microfiche \$0.95)

**References:**

NASA-TN-D-3693 (N66-38750), Investigation of Magnetically Soft, High-Temperature Cobalt-Iron Alloy, and NASA TN-D-4551 (N68-24558), Electrical and Mechanical Properties of a Superior High-Temperature Cobalt-Iron Magnetic Material.

Source: J. P. Barranger  
Lewis Research Center  
(LEW-333)



### COPPER WIRE PLATED WITH NICKEL AND SILVER RESISTS CORROSION

Copper wires are susceptible to galvanic corrosion which originates at breaks in the silver plate where moisture and dissolved gases (oxygen and carbon dioxide) are present. Nickel plating, which has been successfully used in many applications because it cathodically protects the copper wire substrate from galvanic corrosion, is difficult to solder.

To eliminate the existing problems, the copper wire is first plated with 1  $\mu\text{m}$  (40 microinches) of

nickel and then with 1  $\mu\text{m}$  of silver. The double plating combines the corrosion resistance and mechanical protection of nickel with the solderability of silver.

Source: North American Aviation, Inc.  
under contract to  
Marshall Space Flight Center  
(MFS-761)

*Circle 10 on Reader Service Card.*

### RETENTION OF DUCTILITY IN HIGH-STRENGTH STEELS

A process to produce high-strength alloy steel, with retention of ductility, includes a series of steps: tempering, application of stress at an elevated temperature, cooling, and subsequent tempering. The steel is strained at temperatures between 65 and 480°C (150-900°F), cooled, and then retempered. The strain aging at elevated temperatures substantially increases the yield strength (sometimes by as much as 25%) and

favorably shapes the stress-strain curve for good mechanical properties.

Source: V. F. Zackay and E. R. Parker of  
Lawrence Radiation Laboratory  
under contract to  
Argonne National Laboratory  
(ARG-10497)

*No further documentation is available.*

### ETCHING PROCESS MILLS PH 14-8 Mo ALLOY STEEL TO PRECISE TOLERANCE

A chemical milling process was developed that produces finishes on PH 14-8 Mo Alloy to precise tolerances by combining an aqua regia etchant with a sulfonate wetting agent.

The PH 14-8 Mo alloy steel in annealed or heat treated condition is subject to a chemical etchant solution that is held at a temperature of 60 to 62°C (140-145°F). Portions not to be etched are covered with a neoprene-type mask that provides good etch line definition. Metal removal of the exposed surface is at a uniform rate of approximately 0.015 mm (0.6 mil) per minute.

To chemically mill a skin of this alloy to very thin web sections of 0.15 to 0.20 mm (0.006-0.008

in), the skin is placed horizontally with the etching solution in contact with the upper surface only, so that the reaction gases may disperse unimpeded. A dodecylbenzene sulfonic acid wetting agent is used in conjunction with the highly corrosive, aqua regia type etchant solution. This enhances the wettability of the etchant solution and results in a more controlled interfacial reaction between solution and metal surface.

Source: P. W. Mullan and B. L. Chipman of  
North American Rockwell Corp.  
under contract to  
Manned Spacecraft Center  
(MSC-270)

*Circle 11 on Reader Service Card.*

### GAGE OF 6.5% Si-Fe SHEET IS CHEMICALLY REDUCED

The brittleness of 6.5% Si-Fe soft-magnetic alloy sheets or strips prevents their rolling to very thin gages. In the new technique the material is chemically milled to the desired gage after the conventional rolling to safe gage limits.

Either of the two chemical solutions are used to effect the chemical milling of 6.5% Si-Fe strips 30-to-33-cm (12-13 inches) thick. One solution contains equal parts of hydrochloric acid, nitric acid (both technical grades), and water. The other solution is a mixture of equal parts of phosphoric acid and high purity hydrogen peroxide. Each solution effects uniform metal removal. The hydrochloric-acid, nitric-acid solution is more suitable for production purposes

because of its high rate of metal removal, low cost, and ease of storage and handling.

After chemical milling, the remaining material is coated with a zirconia slurry and annealed in a  $3.5$  to  $7.0 \times 10^3$  N/m<sup>2</sup> (0.5-1.0 psi) hydrogen atmosphere at 1,000°C. The surface coating is then removed and the material is quality checked for silicon content, which should be between 6.0 and 6.5%.

Source: D. M. Pavlovic and A. Goldman of  
Westinghouse Electric Corp.  
under contract to  
Manned Spacecraft Center  
(MSC-537)

*Circle 12 on Reader Service Card.*

### GRINDING AS AN APPROACH TO THE PRODUCTION OF HIGH-STRENGTH NICKEL-BASE ALLOYS

In development of dispersion-strengthened nickel-thoria alloys from mechanically comminuted and blended powders, two material compositions have been produced: a 4.0 volume percent thoria in nickel; and a 0.5 weight percent zirconium and 4.0 volume percent thoria in nickel. Nickel, or nickel with 0.5 weight percent zirconium hydride, was ball-milled with added grinding agents for five days to particle sizes ranging from 0.02 to 0.04  $\mu$ m. Four volume percent of colloidal (0.005 to 0.015  $\mu$ m) thoria was then added and the mixture ball-milled for another 24 hours to achieve a random blend. The powder slurry (powder plus liquid grinding agents) was passed through screens, partially dried and compacted. The compacted slabs were then cleaned by heating in hydrogen to a carefully controlled temperature schedule. The cleaned sintered slabs were finally roll-worked in such a way as to promote high temperature strength.

The resulting sheet material specimens had 1095°C

(2000°F) tensile strengths ranging from 11 to  $14 \times 10^7$  N/m<sup>2</sup> (16,000-20,000 psi). Comparable chemically produced commercial sheet products have tensile strengths, at 1095°C, of  $10$  to  $11 \times 10^7$  N/m<sup>2</sup>, or 14,000-16,000 psi. These new alloys can also be produced in bar stock or wire form.

The following documentation may be obtained from:

National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$6.00  
(or microfiche \$0.95)

Reference:

NASA TN-D-5421 (N69-38074), Dispersion - Strengthened Nickel Produced from Ultrafine Comminuted Powders

Source: M. Quatinetz, J. W. Weeton,  
and N. W. Orth  
Lewis Research Center  
(LEW-10515)

### HEAT TREATMENT PROCEDURE TO INCREASE DUCTILITY OF DEGRADED NICKEL ALLOY

A study has been conducted to establish a heat treatment procedure that will increase the room temperature ductility of degraded René 41 in the form of thick plate and forgings. Tests have shown that the room temperature ductility of degraded hardware and out-of-specification mill stock of René 41 can be increased to acceptable values by solution heat treatment of the material at a temperature of 1120 to 1185°C (2050 - 2150°F) for 1 to 2 hours and cooling through a controlled temperature range followed by normal aging in air (16 hours at 760°C, 1400°F). Elongation (a measure of ductility) of test specimens

of rejected forgings tested at room temperature was increased from the original 4% to approximately 13%. (Specification grade René 41 from commercial sources has an elongation of 15 to 20%.) After this heat treatment, the alloy can be subjected to additional heat treatment by the conventional procedures without appreciable decrease in ductility.

Source: M. Prager of  
North American Aviation, Inc.  
under contract to  
Marshall Space Flight Center  
(MFS-12410)

*Circle 13 on Reader Service Card.*

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### NIOBIUM-URANIUM ALLOYS WITH VOIDS OF PREDETERMINED SIZE AND TOTAL VOLUME

To produce a niobium-uranium alloy having voids of predetermined size and total volume, uranium and niobium oxides are mixed with the graphitic carbon in a ball mill and pressed into right-circular tubes having outside and inside diameters of 12 and about 4 mm, respectively. All samples are heated in crucibles within an induction furnace. As the sample is heated and partial vacuum applied to the furnace, the sample temperature is recorded as well as the pressure within the furnace and the amount of gas evolved. After cooling of the sample at the end of each run, several sections perpendicular to the major axis of the tube are taken and examined for porosity by standard metallographic techniques.

While any niobium-uranium alloy may be used,

the eutectic that occurs at about 20% uranium (by weight) is preferred. The stoichiometric gram quantities (28.57 g Nb<sub>2</sub>O<sub>5</sub>, 5.72 g UO<sub>2</sub>, and 7.01 g C) are mixed, pressed, put into tantalum crucibles, and heated to between 1950 and 2100°C. Since the sintering is conducted below the melting point of the alloy, no segregation occurs, and the resultant product is a homogeneous niobium-uranium (20% by weight) alloy containing many voids distributed through the sample.

Source: H. A. Wilhelm and J. K. McCluskey of  
Iowa State University  
under contract to  
Argonne National Laboratory  
(ARG-10490)

*No further documentation is available.*

## SUPERIOR SILVER-BASE TERNARY ALLOY FOR RING LEAD WIRES

Slip-ring lead wires used in guidance and control systems must have high electrical conductivity, a tensile strength of at least  $21 \times 10^7 \text{ N/m}^2$  (30,000 psi), high ductility, and be capable of being soldered and welded. A wire composed of ternary alloys of silver, which includes less than 5% copper plus a minor addition of a third element, provides the following properties:

- (1) Minimum electrical conductivity of 90%; maximum specific resistivity of 1.9 microhms-cm.
- (2) Minimum tensile strength of  $21 \times 10^7 \text{ N/m}^2$ .
- (3) Ability (of 42-gage wire) to withstand at least 400 bends of 90 degrees or more over a 0.6 mm (0.025 in) radius, while supporting a tensile stress of  $41 \times 10^6 \text{ N/m}^2$  (6000 psi).
- (4) Ability to withstand corrosion in fluorine moisture environment.

A satisfactory composition is silver with 1.0% copper and 0.2% nickel. The copper serves primarily to increase strength with only a minor sacrifice in elec-

trical conductivity. The desired range of copper falls between 0.25 and 5.0%. Generally, all the copper is in solid solution.

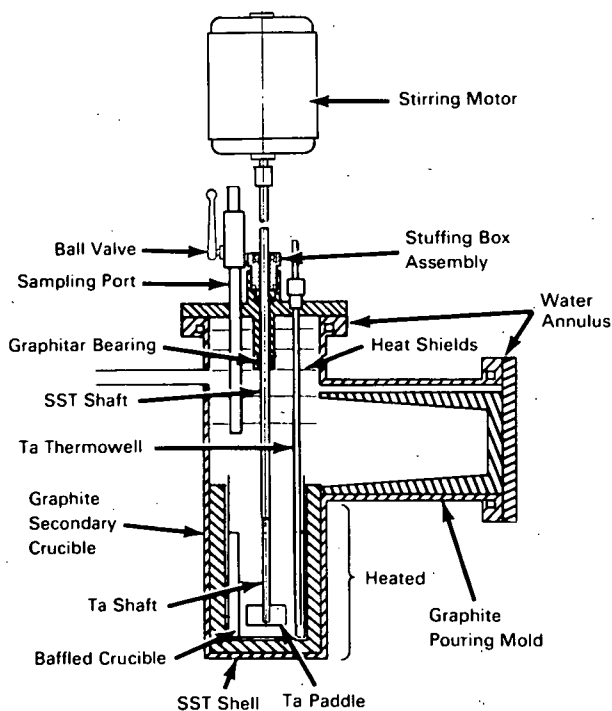
The minor ternary addition serves to increase resistance to heat softening and to raise the recrystallization temperature without significantly decreasing conductivity. In addition to nickel, beryllium is a satisfactory ternary addition. These elements, acting as a dispersed phase, have only slight solubility in solid silver. Ternary element content ranges from 0.1 to 1.0%.

These new alloys are made by melting under hydrogen or by melting with suitable deoxidation and are readily drawn to wire by conventional drawing methods, using intermittent heat-treatment steps. An unexpected advantage of these alloys is their resistance to discoloration on heating in air.

Source: R. H. Ernst and D. N. Williams  
Marshall Space Flight Center  
(MFS-1540)

*Circle 14 on Reader Service Card.*

## PREPARATION OF THORIUM BY MAGNESIUM-ZINC REDUCTION



THORIUM DIOXIDE REDUCTION APPARATUS

The method for commercial production of thorium metal involves metallothermic reduction of thorium tetrafluoride with magnesium zinc alloy in the presence of zinc chloride. The molten salt fluxes are prepared from anhydrous, reagent-grade salts, except for magnesium chloride, which is obtained as a by-product from a process used for the production of zirconium. The thorium dioxide is a high-fired material that had been screened to -325 mesh. The zinc and magnesium were 99.99 and 99.95% pure, respectively.

Although complete reductions were achieved in air, the rate and extent of reduction were improved by the use of argon. The apparatus shown in the figure was used for the argon experiments. It consists primarily of a resistance-heated, tilt-pour, stainless-steel furnace. The reductions were conducted in a tantalum crucible, equipped with four baffles to enhance mixing, and located inside a graphite secondary container. By tilting the entire furnace, the molten contents of the crucible could be poured into the mold.

Under optimum conditions, over 99% reduction of thorium dioxide was obtained in less than 4 hours, with product purities up to 99.7%. These optimum conditions were: 800°C, argon atmosphere, a magnesium concentration of 5 to 10 w/o in the zinc phase, a flux containing a large proportion of magnesium chloride and  $\approx 10$  m/o calcium or magnesium fluoride, and a thorium dioxide flux weight ratio of about 0.22%.

The following documentation may be obtained from:

National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$6.00  
(or microfiche \$0.95)

Reference:

ANL-7058 (N65-36816), Preparation of Metals by Magnesium-Zinc Reduction- Part II Reduction of Thorium Dioxide

Additional documentation is also available from:

U.S. Patent Office

Single document price (\$0.50)

Reference:

U.S. Patent No. 3,164,462, Preparation of Thorium Metal from the Oxide.

Source: A. V. Hariharan, J. B. Knighton  
and R. K. Steunenberg  
Argonne National Laboratory  
(ARG-10245)

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## STRESS CORROSION CRACK INHIBITING METHOD FOR TITANIUM

A method is proposed for the prevention of stress corrosion cracking of high strength titanium alloys under exposure to aqueous and other solvent environments.

The method requires adding a quantity of oxyanions to the liquid in excess of the number of chloride, bromide, or iodide ions present. The oxyanions are generally phosphates, chromates, sulfates, nitrates, or carbonates. Generally, the molar concentration of oxyanion is set from 10 to 100 times higher than the concentration of the halide ions. Since the cation does not affect the results, the salts of potassium, sodium, lithium, or others may be used. Additionally, organic compounds that contain oxyanion groups, such as

nitromethane, may be used in organic solvents.

The proposed method is particularly concerned with the problem of residual chloride, bromide, or iodide ions in titanium metal alloys that are exposed to inert solvents bearing traces of the same halides. Where high concentrations of halide ions (on the order of one molar) are present in the solvents, stress corrosion cracking is difficult to prevent.

Source: T. R. Beck of  
Boeing Corp.  
under contract to  
NASA Pasadena Office  
(NPO-10271)

*Circle 15 on Reader Service Card*

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## GLASS BEAD PEENING TO RETARD STRESS CORROSION FAILURE OF TITANIUM TANKS

To eliminate the failure of titanium rocket-propellant storage tanks as a result of exposure to an  $N_2O_4$  environment, the tanks are subjected to rigidly controlled glass bead peening. Essentially, glass bead peening is a process whereby small glass spheres are forcibly impelled against a surface. In this application, the glass beads ranged in size from 0.15 to 0.25 mm in diameter. The glass beads are sprayed from a four-barreled nozzle held at the end of a

rotatable nozzle arm. Each nozzle discharges about 0.25 million beads per second. The nozzle arm is positioned on the end of a lance arm to permit insertion into the tank. The lance arm is rotatable, and a lance carriage permits the linear positioning of the nozzle. The tank can also be rotated independently. Using combinations of these movements it is possible to program carefully controlled coverage of the interior of the tank and as a result induce a uniform com-

pressive stress in the peened surface to prevent stress corrosion.

The glass bead flow is monitored with the electrostatic charge built up around each of four lengths of Tygon tubing through which the beads flow. The angle of impact and the degree of coverage is con-

trolled by the tank rotation and the movement of the lance and the lance carriage.

Source: C. R. Manning, T. T. Bales,  
W. R. Lisagor, and M. B. Seyffort  
Langley Research Center  
(LAR-319)

*Circle 16 on Reader Service Card.*

## FABRICATION TECHNIQUES DEVELOPED FOR SMALL-DIAMETER THIN-WALL TUNGSTEN AND TUNGSTEN ALLOY TUBING

Small-diameter, thin-wall tubing of nuclear quality may be produced from tungsten or tungsten-25 w/o rhenium (W-25 Re) tube blanks by double extrusion. The starting material, generally a pressed and sintered product, is in the form of a sleeve 3.17 cm outside diameter (o.d.) and 2.54 cm inside diameter (i.d.), and 10.2 cm long. Both initial and reextrusion processes use a filled-billet technique. The resulting tube blanks are 0.64 cm o.d. by 0.51 cm i.d. and 75 cm long, are structurally sound and suitable for secondary fabrication operations.

Secondary fabrication until recently has received little attention. Work has been done on secondary fabrication operations concentrating on the W-25 Re alloy. Plug-drawing has emerged as an excellent technique both for the reduction of the overall tube dimensions and for the removal of the longitudinal surface striations that result from the filled-billet-extru-

sion sequence. Plug-drawing also obviates the need for core removal at the finished-tube size (0.64 mm o.d.).

The following documentation may be obtained from:

National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$6.00  
(or microfiche \$0.95)

Reference:

ANL - 7151 (N67-25067), The Development of Techniques for Fabrication of Small-Diameter Thin-Wall Tungsten and Tungsten-Alloy Tubing

Source: W. R. Burt, D. C. Brillhart,  
R. M. Mayfield, and F. J. Karasek  
Metallurgy Division  
Argonne National Laboratory  
(ARG-10100)

## METHOD FOR CONTROLLING DENSITY AND PERMEABILITY OF SINTERED POWDERED METALS

An improved, relatively low-cost method has been developed to produce porous metals with predetermined pore size, pore spacing, and density utilizing powder-metal processes. This method utilizes sized, hydrogen-reduced, angular tungsten powder.

The desired material is produced as follows:

1. The metal powders of selected size are blended with relatively inert particles that will give pores of the desired size and geometry. The final density is determined principally by the amount of inert material incorporated in the metal powder. In actual practice, flake copper has been utilized as

the inert material and has been blended with angular tungsten powder of 0.8  $\mu\text{m}$  average size.

2. The metal powder mixture is pressed hydrostatically to produce a compact of the desired size and shape.
3. The pressed compact is presintered at a temperature sufficiently high to produce grain growth and densification in the powder mass surrounding each inert particle, but at a temperature below the melting or volatilization temperature of the inert particles.
4. Upon completion of presintering, the temperature

is raised sufficiently to evaporate the pore-forming inert particles and produce the desired densification of the pressed compact.

5. Machining the compact to final dimensions is accomplished by infiltrating the porous compact with copper or the more inert gold, machining to the desired geometry, and reevaporating the infiltrated metal at high temperature in vacuum. Porous compacts of less than 70 percent theoretical density were produced in sizes of up to 10-by-5-by-1.3 cm (4-by-2-by-½ in).

The following documentation may be obtained from:

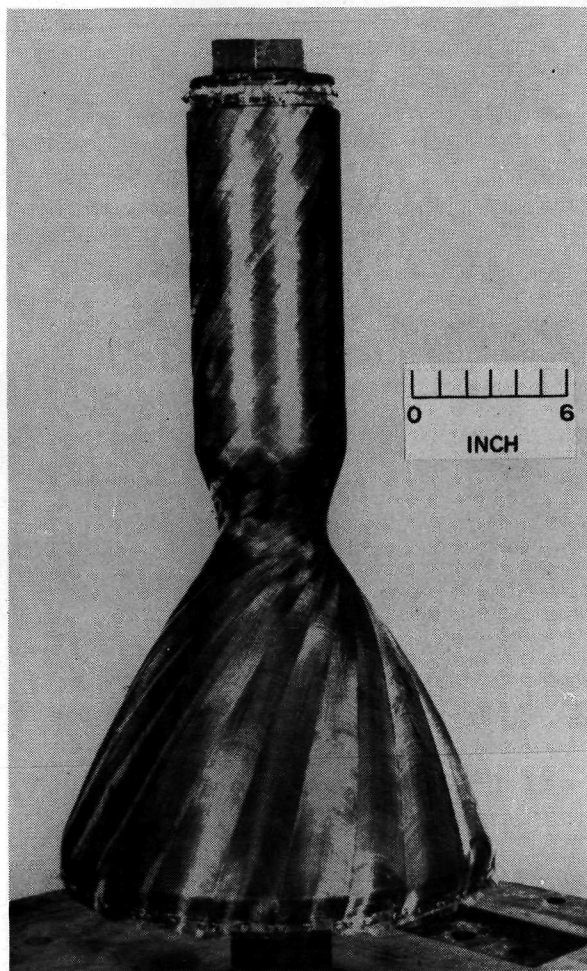
National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$6.00  
(or microfiche \$0.95)

Reference:

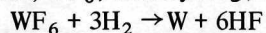
NASA CR-54707 (N66-35050), Development and Testing of Porous Ionizer Materials.

Source: H. H. Todd of  
Electro-Optical Systems, Inc.  
under contract to  
Lewis Research Center  
(LEW-10393)

### FABRICATION OF LARGE TUNGSTEN STRUCTURES BY CHEMICAL VAPOR DEPOSITION



Process details have been developed for the fabrication of a large, pure-tungsten, rocket-motor chamber shell (or similar geometric structure). The process involves the chemical vapor-phase deposition of tungsten, which is accomplished by reducing tungsten hexafluoride,  $WF_6$ , with hydrogen,  $H_2$ :



Metallic tungsten of essentially 100% purity and density is produced in this reaction and is built up as a dense deposit on a heated mandrel assembly.

A shell with dimensions 13.33 cm (5.25 in.) diameter at the small end, 40.6 cm (16 in.) diameter at the nozzle exit, and 61 cm (24 in.) length, is the largest structure of this type known to have been constructed. The shell serves as the liner wall of a regeneratively cooled, hydrogen-fluorine rocket thrust chamber.

The following documentation may be obtained from:

National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$3.00  
(or microfiche \$0.95)

Reference:

NASA CR-72742 (N71-14135), Investigation of Advanced Regenerative Thrust Chamber Designs.

Source: V. R. Stubbs, W. J. Lewis,  
and V. E. Kahle of  
Aerojet-General Corp.  
under contract to  
Lewis Research Center  
(LEW-11239)

## MAGNESIUM-ZINC REDUCTION IS EFFECTIVE IN PREPARATION OF URANIUM, THORIUM, AND PLUTONIUM

The preparation of metals by magnesium-zinc reduction has been studied using uranium oxides, thorium dioxide, and plutonium dioxide as starting materials. In the preparation of uranium, uranium oxide was suspended in a flux composed of alkali and alkaline earth halides. It was then reduced by vigorous mixing with Zn/5Mg alloy at temperatures between 650 and 800°C. The reductions were conducted in an alumina crucible in air. As the uranium metal was produced, it dissolved in the liquid-metal alloy and was later recovered by vacuum distillation. Laboratory-scale (4g of uranium) reduction of  $U_3O_8$  was more than 99% complete in periods as short as 10 minutes. Uranium losses in the process were well below 1%.

In the preparation of thorium, the reduction rates of thorium dioxide were slower than those obtained with  $U_3O_8$ , but the effects of changes in flux composition were similar. A reduction of more than 99% was obtained in less than 4 hours, and product purities of up to 99.7% were achieved. Thorium tetrafluoride is also readily reduced by the magnesium-zinc alloy.

In the preparation of plutonium, the reduction

rate of plutonium dioxide, unlike those of uranium and thorium oxides, was relatively independent of flux composition. The most satisfactory reduction at 800°C was obtained with a magnesium concentration of about 10 wt% in the liquid-metal phase. The use of plutonium dioxide instead of the fluorides as the starting material has the advantage of greatly reducing the neutron-emission hazard resulting from ( $\alpha, n$ ) reactions. Plutonium losses from incomplete reduction were less than 1%.

The following documentation may be obtained from:

National Technical Information Service  
Springfield, Virginia 22151  
Single document price \$6.00  
(or microfiche \$0.95)

### Reference:

ANL-7057 (N65-36130) Part I and (N65-36816)  
Part II, Preparation of Metals by Magnesium-Zinc  
Reduction.

Source: J. B. Knighton and R. K. Steunenberg  
Chemical Engineering Division  
Argonne National Laboratory  
(ARG-10050)

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## IGNITION OF BINARY ALLOYS OF URANIUM

Seventy binary uranium alloys, representing 24 different additives, were studied in an attempt to improve the properties of the parent metal, uranium. Several of these additives were common impurities of uranium; others were selected because of their particular interest to the nuclear field. Most of the alloys were prepared in nominal concentrations of 0.5, 1, and 2 atom percent. All were analyzed for carbon, nitrogen, oxygen, hydrogen, and trace elements as well as for intentionally added constituents.

The experiments were performed by placing nominal 8.5-mm alloy cubes in a flowing oxidizing atmosphere within a furnace. The temperature of the furnace was then increased 10°C per minute. Differences in the sample temperature-time curves were noted for the various binary uranium alloys. Results were interpreted in terms of the effect of the alloying addition on the previously demonstrated transition of the oxide at 400°C from an autocatalytic to a protective form.



## SPECIFIC METALS

Additions of aluminum and certain other alloys, including beryllium, bismuth, carbon, lead, molybdenum, niobium, palladium, platinum, ruthenium, silicon, titanium, and vanadium, inhibited the oxide transition and thereby lowered the ignition temperature. Another effect produced notably by copper (but also by bismuth, lead, palladium, ruthenium, and vanadium) was an increase in the protectiveness of the oxide formed at temperatures above 500°C. Very complex temperature-time curves were obtained when both effects occurred with the same alloy.

Alloying additions of cerium, chromium, hydrogen, iron, nickel, rhodium, silver, tantalum, thorium, and zirconium had no significant effect on the ignition behavior of uranium at the low concentration levels studied.

Source: J. G. Schnizlein, L. Baker, Jr.,  
and J. D. Bingle of  
Chemical Engineering Division  
Argonne National Laboratory  
(ARG-10057)

*No further documentation is available.*

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## INCREASED THERMAL SHOCK RESISTANCE OF CERAMIC COATING

To increase the thermal shock resistance of a plasma-arc-sprayed coating of zirconia used as a heat barrier on a metal substrate, a small amount of copper is deposited on the granules of the zirconia powder before it is arc-sprayed onto the substrate. The thermal shock resistance of the finished coating is increased by the dispersed copper phase between the boundaries of the zirconia granules. A composite powder consisting of zirconia with a dispersion of

iridium can be plasma-arc-sprayed to provide a coating that maintains its structural integrity at temperatures up to 2200°C (4000°F) in an oxidation atmosphere.

Source: H. W. Carpenter of  
North American Aviation, Inc.  
under contract to  
Marshall Space Flight Center  
(MFS-1862)

*Circle 17 on Reader Service Card.*

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